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Precipitation Reactions in 8090 SiC Particulate Reinforced MMC

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SUMMARY

Microhardness testing, differential scanning calorimetry and transmission electron microscopy have been used to study the precipitation reactions in the matrix of 8090-SiC particulate reinforced metal matrix composite. Unreinforced 8090 produced via the powder route and also by ingot metallurgy was similarly studied for comparison. The 8090 MMC matrix exhibited a more rapid age hardening response than the unreinforced alloy, but this accelerated ageing cannot be attributed to enhanced δ' or S' precipitation. It is suggested that rapid formation of GPB zones or increased dislocation interaction may be responsible for this effect.

Departmental Reference: Materials/Structures 291

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1 INTRODUCTION

There is currently much interest in SiC particulate reinforcement of aluminium-lithium based alloys such as 8090 (Al-Li-Cu-Mg) to provide a light-weight, high stiffness and isotropic metal matrix composite (MMC)[1]. The composite materials may be heat-treated to develop an optimum balance of mechanical properties. 8090/SiC MMCs produced via spray deposition are reported to exhibit similar ageing kinetics to those of the matrix alloy[2], but similar materials produced via solid state powder processing and incorporating a higher volume fraction of particulate reinforcement have been shown to exhibit a more rapid ageing response than the unreinforced 8090 matrix[3]. The present research set out to investigate the microstructural changes during age hardening of SiC particulate reinforced 8090 in order to provide a better understanding of the ageing kinetics and thereby complement the efforts towards property optimisation. (JS)

2 EXPERIMENTAL PROCEDURE

8090 (Al-2.3Li-1.2Cu-0.7Mg-0.1Zr) MMC containing 20 wt% SiC (~ 3μm) was manufactured from powders by BP Research Centre at Sunbury and processed at RAE Farnborough to 1.6 mm sheet. Unreinforced 8090 produced via powder (PM) and ingot metallurgy (IM) were studied for comparison. The wrought materials were solution treated (560°C for MMC; 530°C for IM and PM) and water quenched prior to age hardening. The ageing response of the MMC matrix, exposed by polishing away surface SiC, was determined via microhardness testing. Differential Scanning Calorimetry (DSC) was undertaken at a heating rate of 10°C/min up to 450°C. Specimens were prepared for TEM by electrolytic polishing and were examined in a JEOL 200CX STEM. A Cambridge electron probe microanalyser (Microscan 9) was used for microanalysis.

3 RESULTS

The natural ageing (NA) curves (Fig 1) of the IM and PM alloys are similar in shape and are consistent with previously established natural ageing responses for 8090 IM sheet[4]. The PM material is seen to be inherently harder than the IM alloy. The hardness of the As Quenched (AQ) MMC matrix is high and a plateau is reached after 10 hours of natural ageing, but the hardness increment during NA is small compared to that of the unreinforced alloys. Artificial Ageing (AA) responses are similar for the IM and PM alloys at 170°C (Fig 2). The curves are characterised by an increasing hardness up to a peak at ≈ 50 hours. By contrast the MMC matrix reached a peak hardness (greater than that of the unreinforced PM alloy) after 5 to 6 hours.

DSC curves for the PM and IM alloys were identical. For all heat treatment conditions endotherms at $\simeq 245°C$, corresponding to δ' dissolution, and exotherms at $\simeq 320°C$ associated with S' precipitation were observed (Fig 3a). In AQ and NA conditions the exotherm at $\simeq 180°C$ can be attributed to δ' precipitation. The exotherm at $\simeq 104°C$ and the endotherm at $\simeq 150°C$ could correspond to the formation and dissolution of GPB zones[5]. The GPB zone formation reaction was absent after natural ageing. Results from the MMC material show the δ' dissolution and S' precipitation reactions to be at the lower temperatures of $\simeq 230°C$ and $\simeq 280°C$ respectively (Fig 3b). In the AQ and NA conditions the endotherm associated with δ' precipitation in the MMC is at the higher temperasture of $\simeq 195°C$. No exothermic reaction corresponding to GPB zone formation was present in AQ MMC specimens.

The PM and MMC materials exhibit numerous dispersoids, examples of which are shown in Fig 4. X-ray analysis of the particles in TEM gave peaks for Al and Cu only, the peak for Cu generally being smaller than for the matrix (*ie* they contain Al and light elements such as Li, O and C only). The diffraction patterns shown in Fig 5 were consistent with reflections from Li_2CO_3 and $LiAlO_2$ respectively. The dispersoids are unaffected by heat treatment but they are lithium rich and as a consequence a δ' PFZ develops around the dispersoids during ageing. However, the presence of the dispersoid particles has no effect on the ageing kinetics of 8090 PM or 8090 MMC.

TEM studies showed that the AQ MMC has a high density of dislocations adjacent to the SiC particles with few helical dislocations being observed. The AQ distribution of δ' was similar in MMC and unreinforced 8090 with an average diameter of 3-5 nm. After artificial ageing δ' was finer in the MMC (*eg* 20 nm compared to 24 nm after 16 hours at 190°C); this observation has been confirmed by small angle neutron scattering (SANS) studies[6]. Microanalysis of the matrix and backscattered imaging of MMC indicated that any interaction between the SiC particles and the alloy matrix was limited, furthermore there was no evidence of additional precipitation of δ' PFZs adjacent to SiC particles. In all the 8090 based materials no significant S' was evident after ageing at 170°C for 7 hours *ie* just exceeding peak strength of the MMC matrix. After ageing at 190°C for 16 hours (corresponding to peak strength of unreinforced 8090), the laths of heterogeneously nucleated S' in both 8090 PM and MMC materials were of similar length (Fig 6). Extended artificial ageing at 190°C for 48 hours resulted in extensive precipitation of S', the higher dislocation density within the MMC giving rise to more widespread heterogeneous nucleation of S'.

4 DISCUSSION

The presence of lithium-containing oxides and carbonates has been established; these compounds have been identified by X-ray diffraction to be the main constituents in the surface oxide layer of sheet products of Al-Li alloys[7]. They will be formed on the surface of the 8090 alloy powder particles after atomisation, and during fabrication must be disrupted to form the dispersoid particles seen in the final product. Interaction of the dispersoids with the dislocations generated during heat treatment has been observed, and the dispersoid particles are likely to have a strengthening effect: it is believed that the presence of dispersoids contributes to the higher strength of the PM 8090 compared to IM 8090. The results clearly show that the age-hardening of the matrix is more rapid for MMCs than for unreinforced 8090. This is consistent with earlier work which showed that maximum strength of the composite was achieved after 3 hours at 170°C [3]. The more rapid ageing response has previously been attributed to additional heterogeneous precipitation of S' on dislocations within 8090 MMC, but in the present microstructural study no such S' was observed after ageing treatments corresponding to the peak strength condition, indicating that precipitation of S' alone is not responsible for the rapid age-hardening of the matrix.

In the AQ condition the distribution and size of the very fine δ' particles was essentially the same for IM, PM and MMC materials; this suggests that the nucleation of δ' is unaffected by the presence of SiC. After artificial ageing the observation of finer δ' within the MMC matrix compared with 8090 IM/PM indicates that growth of δ' is retarded. This could be due to lithium incorporation into the SiC and dispersoid particles thus depleting the concentration of the solute in the matrix. However there were no obvious δ' free zones adjacent to the SiC particles suggesting that any such reaction is limited. Furthermore, δ' free zones around dispersoid particles in the MMC are also present in the PM material within which the size of the δ' particles are unaffected. Thus the retarded growth of δ' in MMCs probably results from slower diffusion of lithium solute to the δ' nuclei, which may be caused by a lower vacancy concentration. The presence of Si (from the SiC) in the matrix will lower the concentration of free vacancies on account of the high Si atom to vacancy binding energy. However there was no evidence for Si release into the matrix, and any such reaction would retard all precipitation reactions contrary to the present observations of S' precipitation.

It is clear that the rapid hardening response of the MMC matrix must be due to some dislocation interaction other than that associated with δ' or S'

precipitates. The absence of a low temperature exotherm in the DSC trace for AQ MMC suggests that GPB zones, or some other precursor to S' , may be present within the AQ MMC matrix and lead to rapid age hardening; however, it must be stressed that there is no direct evidence (*eg* electron diffraction) for the existence of such zones. Furthermore, during heat-treatment considerable stresses are built up within the MMC as a consequence of the mismatch of thermal expansion between SiC and matrix (*eg* 350 MPa at 200°C). The consequent plastic deformation within the matrix may lead to additional interaction between mobile dislocations and solute atoms, vacancies or between dislocations themselves. The contributions of these interactions to the age-hardening behaviour is currently being assessed. The rapid overageing behaviour of 2124 MMC[1] may be associated with similar mechanisms.

5 CONCLUSIONS

Studies of the age-hardening characteristics of 8090 reinforced with 20% SiC has led to the following conclusions:

(1) The 8090 MMC matrix material exhibits a more rapid age-hardening than the unreinforced alloy.

(2) The presence of lithium-containing oxides and carbonates has been identified, but did not influence the ageing kinetics.

(3) The accelerated ageing cannot be attributed to enhanced δ' or S' precipitation. It is suggested that rapid information of GPB zones or increased dislocation interaction may be responsible for this effect.

Acknowledgment

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REFERENCES

No.	Author	Title etc
1	C.J. Peel R. Moreton S.J. Flitcroft	Progress towards the optimisation of particulate reinforced composites for aerospace applications. Proc. Conf. 'Metal Matrix Composites: Property Optimisation and Applications'. Inst of Metals, London p.4.1 (1989)
2	J. White I.G. Palmer I.R. Hughes S.A. Court	Development of metal matrix composite systems based on aluminium-lithium. Proc. Conf. 'Aluminium-Lithium Alloys 5'. Williamsburg, Virginia, p 1635 (1989)
3	W.S. Miller L.A. Lenssen F.J. Humphreys	The strength, toughness and fracture behaviour in aluminium-lithium based metal matrix composites. *ibid*, p 931, (1989)
4	H.M. Flower P.J. Gregson C.N.J. Tite A.K. Mukhopadhyay	The effect of composition and heat treatment upon the microstructure/property relationships in Al-Li-Cu-Mg alloys. Proc. Conf. 'Aluminium Alloys - their physical and mechanical properties' EMAS, p 743 (1986)
5	A.K. Mukhopadhyay C.N.J. Tite H.M. Flower P.J. Gregson F.R. Sale	Thermal analysis study of the precipitation reactions in Al-Li-Cu-Mg-Zr alloys. Proc. Conf. 'Aluminium-Lithium alloys 4', Les Editions de Physique, Paris, p 439 (1987)
6	R.J. Stewart	Unpublished work, Reading University
7	P. Holdway A.W. Bowen	The measurement of lithium depletion in aluminium-lithium alloys using X-ray diffraction. *J. Mat. Science*, **24**, p 3841 (1989)

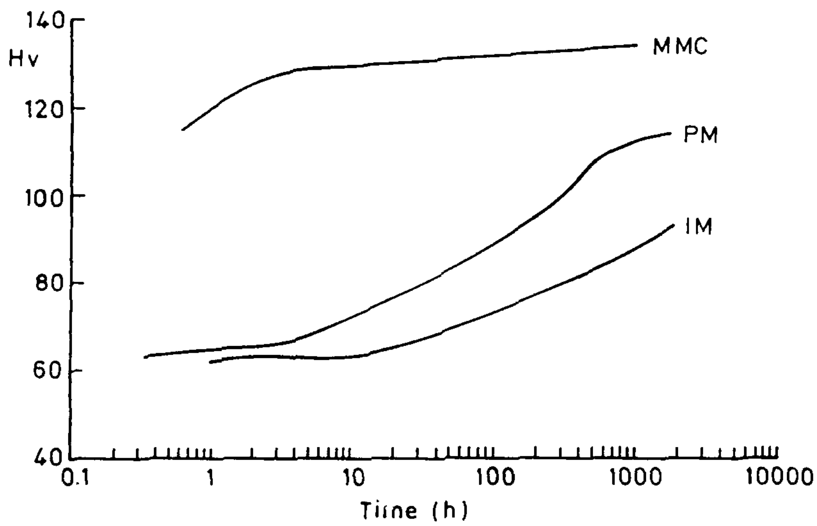


Fig 1 Natural ageing curves for 8090 PM, IM and MMC matrix materials

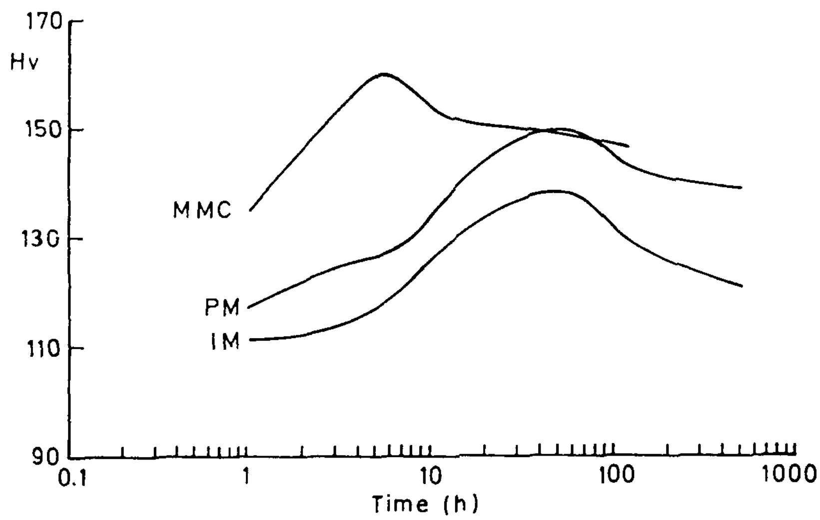


Fig 2 Ageing curves (170°C) for 8090 PM, IM and MMC matrix materials

Fig 3

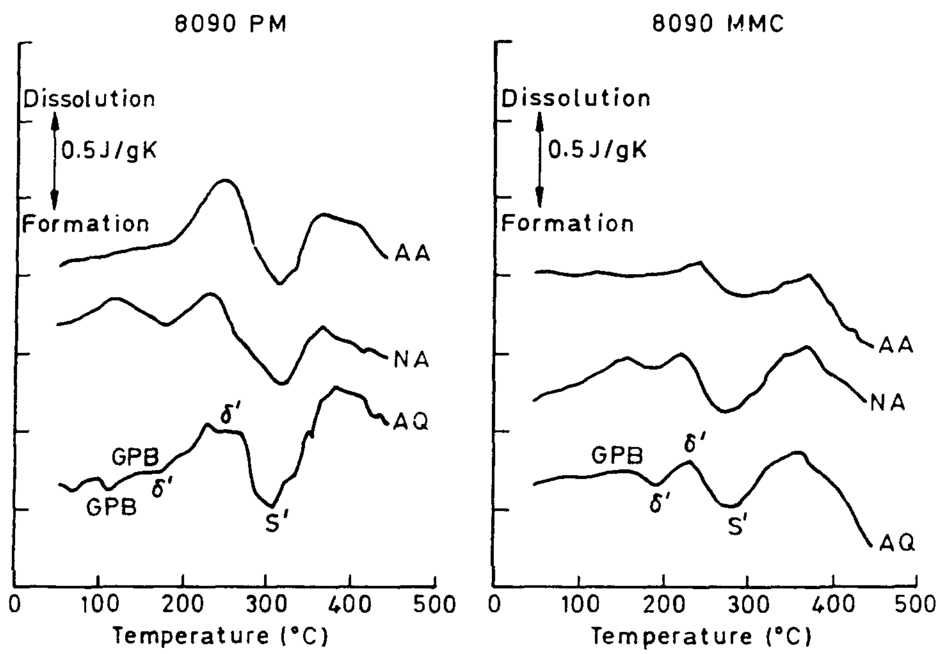


Fig 3 Specific heat capacity curves for 8090 PM, IM and 8090 MMC materials in the as-quenched (AQ), naturally aged (NA) and artificially aged (190°C/16h) conditions

Fig 4 TEM micrograph of dispersoid particles in 8090 PM material

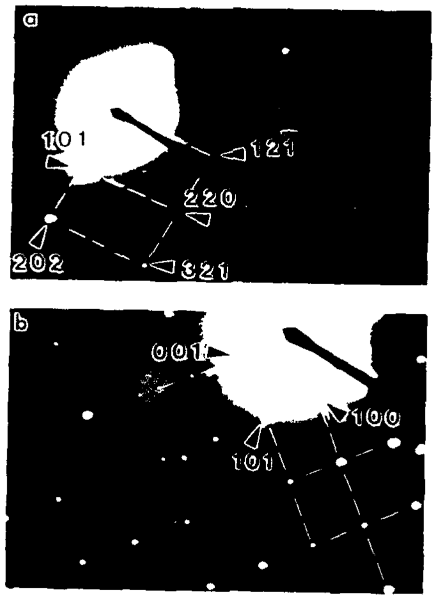
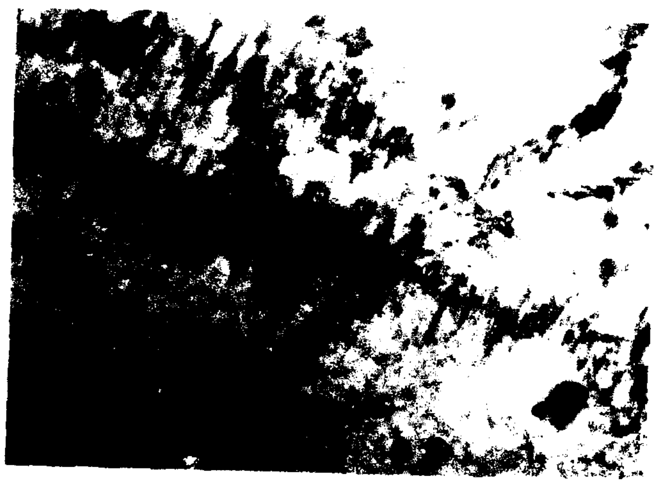


Fig 5 Electron diffraction patterns from
(a) Li_2CO_3 ([$\bar{1}$11] beam direction) and
(b) γ-$LiAlO_2$([010] beam direction)

Fig 6a&b

(a)

0.5 μm



(b)

Fig 6 TEM micrographs of
(a) 8090 PM and
(b) 8090 MMC aged at 190 C for 16h

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17. Abstract

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